

## CERTIFICATE OF ANALYSIS

December 30, 2002

Bechtel Corporation 362 Injun Hollow Road East Hampton, CT 06424

Attention: Alan Heter

#### **AMENDED**

SDG Number : 21324

Date SDG Received : October 15, 2002

Number of Samples : Six (6)
Sample Type : Soil

Data deliverable : Summary Report

## I. Introduction

On October 15, 2002, six soil samples were received at STL Richland (STLR) for radiochemical analysis on four chain-of-custodies (2002-00031,2002-00035, 2002-0039, 2002-0041). Upon receipt, the samples were assigned to Lot Number J2J160129 with the laboratory ID numbers to correspond with the Bechtel Corp. (BCT) specific IDs as listed on the cover page.

## II. Analytical Results/Methodology

The analytical results for this report are presented by laboratory sample ID. Each set of data includes sample identification information; analytical results and the appropriate associated statistical errors.

The requested analyses are:

Alpha Spectroscopy

Americium/Curium-isotopic by method RICH-RC-5080 Plutonium-238, 239/240 by method RICH-RC-5010

Gas Proportional Counting

Strontium-90 by method RICH-RC-5006

Gamma Spectroscopy

Gamma Spec by method RICH-RC-5017

Liquid Scintillation Counting Tritium by method RICH-RC-5037

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> Technetium-99 by method RICH-RC-5065 Carbon-14 by method RICH-RC-5022 Iron-55 by method RICH-RC-5074 Nickel-63 by method RICH-RC-5069 Plutonium-241 by method RICH-RC-5010

# III. Quality Control

The analytical results for each analysis performed under SDG 21324 includes a minimum of one Laboratory Control Sample (LCS) and one method (reagent) blank and one duplicate sample analysis. Any exceptions have been noted in the "Comments" section.

QC and sample results are reported in the same units.

#### IV. Comments

#### Alpha Spectroscopy

### Americium/Curium-isotopic by method RICH-RC-5080:

All results are below the CRDL and/or below the MDC. Except as noted, the LCS, sample duplicate, batch blank and sample results are within contractual requirements.

Sample 9528-0002-019F failed with low tracer recovery (COC 2002-00035) in the first batch analysis. In the reanalysis batch, the result is below the CRDL and the MDC. The LCS, sample duplicate, batch blank and sample results are within contractual requirements.

# Plutonium-238, 239/240 by method RICH-RC-5010:

All results are below the CRDL and/or below the MDC. The LCS, sample duplicate, batch blank and sample results are within contractual requirements.

#### Gas Proportional Counting

#### Strontium-90 by method RICH-RC-5006:

The MDA is slightly greater than the CRDL for sample 9528-0004-010F and it's duplicate due to matrix effects (these samples were processed twice). All other results are below the CRDL and/or below the MDC. Except as noted, the LCS, sample duplicate, batch blank and sample results are within contractual requirements.

### Gamma Spectroscopy

#### Gamma Spec by method RICH-RC-5017:

Seventeen of the eighteen requested radionuclides have been reported for each sample. The Bi-212 was over eight half lives since collection, therefore, not reported. Except as noted, the LCS, sample duplicate, batch blank and sample results are within required limits.

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## Liquid Scintillation Counting

### Tritium by method RICH-RC-5007:

All results are below the CRDL and/or below the MDC. The LCS, sample duplicate, batch blank and sample results are within contractual requirements.

#### Technetium-99 by method RICH-RC-5065:

All results are below the CRDL and/or below the MDC. The LCS, sample duplicate, batch blank, matrix spike, and sample results are within contractual requirements.

## Carbon-14 by method RICH-RC-5022:

All results are below the CRDL and below the MDC. The LCS, sample duplicate, batch blank and sample results are within contractual requirements.

#### <u>Iron-55</u> by method RICH-RC-5074:

All results are below the CRDL and below the MDC. The matrix spike is at 45% and the LCS is at 28%. Since all results are well below the CRDL, the data are accepted as is. Except as noted, the LCS, sample duplicate, batch blank, matrix spike, and sample results are within contractual requirements.

#### Nickel-63 by method RICH-RC-5069:

All results are below the CRDL and/or below the MDC. The LCS, sample duplicate, batch blank, matrix spike, and sample results are within contractual requirements.

# Plutonium-241 by method RICH-RC-5080:

All results are below the CRDL and below the MDC. The LCS, sample duplicate, batch blank and sample results are within contractual requirements.

I certify that this Certificate of Analysis is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager, or a designee as verified by the following signature.

Reviewed and approved:

Barbara M. Gillespie Project Manager

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# **Drinking Water Method Cross References**

	DRINKING WAT	ER ASTM METHOD CROSS REFERENCES
Referenced Method	Isotope(s)	STL Richland's SOP number
EPA 901.1	Cs-134, I-131	RICH-RC-5017
EPA 900.0	Alpha & Beta	RICH-RC-5014
EPA 903.1	Ra-226	RICH-RC-5005
EPA 904.0	Ra-228	RICH-RC-5005
EPA 905.0	Sr89/90	RICH-RC-5006
ASTM D2460	Total Radium	RICH-RC-5027
Standard Method 7500-U-C & ASTM D5174	Uranium	RICH-RC-5058
EPA 906.0	Tritium	RICH-RC-5007
NOTE:		
The Gross Alpha LCS is prepared with Am-24		
The Gross Beta LCS is prepared with Sr/Y-90	(unless otherwise	specified in the case narrative)

## **Uncertainty Estimation**

STL Richland has adopted the internationally accepted approach to estimating uncertainties described in "NIST Technical Note 1297, 1994 Edition". The approach, "Law of Propagation of Errors", involves the identification of all variables in an analytical method which are used to derive a result. These variables are related to the analytical result (R) by some functional relationship, R = constants \* f(x.y.z...). The components (x,y,z) are evaluated to determine their contribution to the overall method uncertainty. The individual component uncertainties (u<sub>i</sub>) are then combined using a statistical model that provides the most probable overall uncertainty value. All component uncertainties are categorized as type A, evaluated by statistical methods, or type B, evaluated by other means. Uncertainties not included in the components, such as sample homogeneity, are combined with the component uncertainty as the square root of the sum-of-the-squares of the individual uncertainties. The uncertainty associated with the derived result is the combined uncertainty (u<sub>c</sub>) multiplied by the coverage factor (1,2, or 3).

When three or more sample replicates are used to derive the analytical result, the type A uncertainty is the standard deviation of the mean value (S/vn), where S is the standard deviation of the derived results. The type B uncertainties are all other random or non-random components that are not included in the standard deviation.

The derivation of the general "Law of Propagation of Errors" equations and specific example are available on request.

**Report Definitions** 

	Report Definitions
Action Lev	An agreed upon activity level used to trigger some action when the final result is greater than or equal to the Action Level. Often the Action Level is related to the Decision Limit.
Batch	The QC preparation batch number that relates laboratory samples to QC samples that were prepared and analyzed together.
Bias	Defined by the equation (Result/Expected)-1 as defined by ANSI N13.30.
COC No	Chain of Custody Number assigned by the Client or STL Richland.
Count Error (#s)	Poisson counting statistics of the gross sample count and background. The uncertainty is absolute and in the same units as the result. For Liquid Scintillation Counting (LSC) the batch blank count is the background.
Total Uncert (#s)  u <sub>c</sub> _Combined  Uncertainty.	All known uncertainties associated with the preparation and analysis of the sample are propagated to give a measure of the uncertainty associated with the result, $u_c$ the combined uncertainty. The uncertainty is absolute and in the same units as the result.
(#s), Coverage Factor	The coverage factor defines the width of the confidence interval, 1, 2 or 3 standard deviations.
CRDL (RL)	Contractual Required Detection Limit as defined in the Client's Statement Of Work or STL Richland "default" nominal detection limit. Often referred to the reporting level (RL)
Le	Decision Level based on instrument background or blank, adjusted by the Efficiency, Chemical Yield, and Volume associated with the sample. The Type 1 error probability is approximately 5%. Lc=(1.645 * Sqrt(2*(BkgrndCnt/BkgrndCnt/Min)/SCntMin)) * (ConvFct/(Eff*Yld*Abn*Vol) * IngrFct). For LSC methods the batch blank is used as a measure of the background variability. Lc cannot be calculated when the background count is zero.
Lot-Sample No	The number assigned by the LIMS software to track samples received on the same day for a given client. The sample number is a sequential number assigned to each sample in the Lot.
MDC MDA	Detection Level based on instrument background or blank, adjusted by the Efficiency, Chemical Yield, and Volume with a Type I and II error probability of approximately 5%. MDC = (4.65 * Sqrt((BkgrndCnt/BkgrndCntMin)/SCntMin) + 2.71/SCntMin) * (ConvFct/(Eff * Yld * Abn * Vol) * IngrFct). For LSC methods the batch blank is used as a measure of the background variability.
Primary Detector	The instrument identifier associated with the analysis of the sample aliquot.
Ratio U-234/U-238	The U-234 result divided by the U-238 result. The U-234/U-238 ratio for natural uranium in NIST SRM 4321C is 1.038.
Rst/MDC	Ratio of the Result to the MDC. A value greater than 1 may indicate activity above background at a high level of confidence. Caution should be used when applying this factor and it should be used in concert with the qualifiers associated with the result.
Rst/TotUcert	Ratio of the Result to the Total Uncertainty. If the uncertainty has a coverage factor of 2 a value greater than 1 may indicate activity above background at approximately the 95% level of confidence assuming a two-sided confidence interval. Caution should be used when applying this factor and it should be used in concert with the qualifiers associated with the result.
Report DB No	Sample Identifier used by the report system. The number is based upon the first five digits of the Work Order Number.
RER	The equation Replicate Error Ratio = $(S-D)/\{sqrt(TPUs^2 + TPUd^2)\}$ as defined by ICPT BOA where S is the original sample result. D is the result of the duplicate, TPUs is the total uncertainty of the original sample and TPUd is the total uncertainty of the duplicate sample.
SDG	Sample Delivery Group Number assigned by the Client or assigned by STL Richland upon sample receipt.
Sum Rpt Alpha Spec Rst(s)	The sum of the reported alpha spec results for tests derived from the same sample excluding duplicate result where the results are in the same units.
Work Order	The LIMS software assign test specific identifier.
Yield	The recovery of the tracer added to the sample such as Pu-242 used to trace a Pu-239/40 method.

Date: 30-Dec-02

# Sample Results Summary STL Richland STLRL

Ordered by Client Sample ID, Batch No.

Report No.: 21084

SDG No: 21324

Client ID	Work Order Number	Parameter	Result +- Uncertainty ( 2s)	Qual	Units	Yield	MDC[MDA	RER
9528-0002-019F	E94PC1AD	TECHNETIUM-99	4.92E-02 +- 2.1E-01	U	pCi/g	100.00%	3.15E-01	
9528-0002-019F	E94PC1AC	CARBON-14	1.39E-02 +- 7.7E-02	U	pCi/g	100.00%	1.44E-01	
9528-0002-019F	E94PC1AJ	NICKEL-63	1.49E+00 +- 5.2E+00	U	pCi/g	83.04%	6.66E+00	
9528-0002-019F	E94PC1AK	IRON-55	-1.76E+01 +- 1.1E+01	U	pCi/g	69.87%	2.47E+01	
9528-0002-019F DUP	E94PC1AQ	CARBON-14	4,73E-02 +- 7.8E-02	υ	pCi/g	100.00%	1.44E-01	0.6
9528-0003-004F	E94PK1AG	PLUTONIUM-238	-9.56E-04 +- 1.9E-03	U	pCi/g	70.40%	2.29E-02	
		PLUTONIUM- 239/40	1.91E-02 +- 1.9E-02	J	pCi/g	70.40%	1.29E-02	
9528-0003-004F	E94PK1AH	PLUTONIUM-241	6.94E-01 +- 3.9E-01	U	pCi/g	70.40%	7.51E-01	
9528-0003-004F	E94PK1AN	ACTINIUM-228	1.33E+00 +- 3.4E-01		pCi/g		2.30E-01	
		AG-108M	-8.03E-03 +- 3.4E-02	U	pCi/g		5.75E-02	
		AMERICIUM-241	1.16E-02 +- 1.5E-01	U	pCi/g		2.19E-01	
		BISMUTH-214	1.35E+00 +- 2.5E-01		pCi/g		1.26E-01	
		COBALT-60	-1.37E-02 + 4.2E-02	U	pCi/g		7.40E-02	
		CESIUM-134	3.33E-02 + 5.0E-02	υ	pCi/g		9.44E-02	
		CESIUM-137	5.62E-01 + 1.2E-01		pCi/g		7.25E-02	
		EUROPIUM-152	-3.53E-02 + 1.1E-01	U	pCi/g		1.82E-01	
		EUROPIUM-154	-2.13E-01 +- 1.4E-01	U	pCi/g		2.02E-01	
		EUROPIUM-155	-1.43E-02 +- 1.1E-01	ប	pCi/g		1.81E-01	
		POTASSIUM-40	1.05E+01 +- 2.0E+00		pCi/g		7.48E-01	
		MAGNESIUM-54	4.25E-02 +- 5.3E-02	U	pCi/g		1.02E-01	
		NIOBIUM-94	8.34E-04 +- 3.8E-02	U	pCi/g		6.87E-02	
		LEAD-212	1.29E+00 +- 2.5E-01		pCi/g		1.07E-01	
		LEAD-214	1.35E+00 +- 2.8E-01		pCi/g		1,35E-01	
		RADIUM-226	1.35E+00 +- 2.5E-01		pCi/g		1.26E-01	
		THALLIUM-208	4.10E-01 +- 9.1E-02		pCi/g		5.31E-02	
9528-0003-004F	E94PK1AL	TRITIUM	2.80E-02 +- 2.2E-02	υ	pCi/g	100.00%	4.85E-02	
9528-0003-004F	E94PK1AD	TECHNETIUM-99	1.27E-01 + 2.1E-01	U	pCi/g	100.00%	3.09E-01	
9528-0003-004F	E94PK1AC	CARBON-14	9.07E-03 +- 7.7E-02	U	pCi/g	100.00%	1.44E-01	
9528-0003-004F	E94PK1AM	AMERICIUM-241	4.79E-03 +- 9.6E-03	U	pCi/g	88.70%	1.30E-02	
		CURIUM-242	0.00E+00 +- 2.1E-02	U	pCi/g	88.70%	2.29E-02	
		CURIUM-243/244	-1.94E-03 +- 2.8E-03	U	pCi/g	88.70%	2.75E-02	
9528-0003-004F	E94PK1AJ	NICKEL-63	2.20E+00 +- 5.9E+00	U	pCi/g	74.39%	7.23E+00	

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RER - Replicate Error Ratio = (S-D)/[sqrt(sq(TPUs)+sq(TPUd))] as defined by ICPT BOA.

rptSTLRchSaSum V3.97 A97 J Qual - No U qualifier has been assigned and the result is below the Reporting Limit, RL (CRDL) or Report Value is Estimated. U Qual - Analyzed for, but the result is less than the Mdc/Mda|Total Uncert or gamma scan software did not identify the nuclide.

# Date: 30-Dec-02

# Sample Results Summary STL Richland STLRL

Ordered by Client Sample ID, Batch No.

Report No.: 21084

SDG No: 21324

Client ID	Work Order Number	Parameter	Result +- Uncertainty ( 2s)	Qual	Units	Yield	MDC MDA	RER
9528-0003-004F	E94PK1AK	IRON-55	-2.26E+01 +- 9.1E+00	U	pCi/g	76.73%	1.63E+01	
9528-0003-004F	E94PK2AF	STRONTIUM-90	1.76E-02 +- 5.4E-02	U	pCi/g	15.21%	1.06E-01	
9528-0003-004F DUP	E94PK1AP	AMERICIUM-241	0.00E+00 +- 1.3E-02	υ	pCi/g	76.04%	1.47E-02	0.6
		CURIUM-242	0.00E+00 +- 2.4E-02	U	pCi/g	76.04%	2.60E-02	0.0
		CURIUM-243/244	0.00E+00 +- 1.3E-02	U	pCi/g	76.04%	1.49E-02	0.3
9528-0003-019F	E94PN1AG	PLUTONIUM-238	4.57E-03 +- 9.2E-03	U	pCi/g	73.45%	1.24E-02	
		PLUTONIUM- 239/40	-9.12E-04 +- 1.8E-03	U	pCi/g	73.45%	2.18E-02	
9528-0003-019F	E94PN1AH	PLUTONIUM-241	2.80E-01 +- 3.6E-01	U	pCi/g	73.40%	7.19E-01	
9528-0003-019F	E94PN1AN	ACTINIUM-228	7.84E-01 +- 2.2E-01	U	pCi/g		4.10E-01	
		AG-108M	1.75E-02 +- 2.8E-02	U	pCi/g		5.12E-02	
		AMERICIUM-241	3.14E-02 +- 1.1E-01	U	pCi/g		1.88E-01	
		BISMUTH-214	4.60E-01 +- 1.8E-01	U	pCi/g		2.37E-01	
		COBALT-60	1,50E-01 +- 5.5E-02	υ	pCi/g		1.18E-01	
		CESIUM-134	1.02E-02 +- 4.1E-02	U	pCi/g		7.67E-02	
		CESIUM-137	2.91E-01 +- 7.5E-02	J	pCi/g		6.78E-02	
		EUROPIUM-152	-2.92E-02 +- 8.6E-02	U	pCi/g		1.44E-01	
		EUROPIUM-154	1.58E-02 + 1.1E-01	IJ	pCi/g		2.08E-01	
		EUROPIUM-155	1.09E-01 +- 9.0E-02	U	pCi/g		1.66E-01	
		POTASSIUM-40	1.28E+01 +- 2.0E+00		pCi/g		4.05E-01	
		MAGNESIUM-54	-8.74E-03 +- 4.0E-02	υ	pCi/g		7.16E-02	
		NIOBIUM-94	1.30E-02 + 3.1E-02	U	pCVg		5.92E-02	
		LEAD-212	5.75E-01 + 1.5E-01		pCl/g		1.09E-01	
		LEAD-214	3.27E-01 +- 2.0E-01		pCi/g		1.11E-01	
		RADIUM-226	4.60E-01 +- 1.8E-01	U	pCi/g		2.37E-01	
		THALLIUM-208	2.75E-01 +- 7.8E-02	U	pCi/g		1.17E-01	
9528-0003-019F	E94PN1AL	TRITIUM	6.05E-02 +- 3.6E-02	υ	pCi/g	100.00%	7.95E-02	
9528-0003-019F	E94PN1AD	TECHNETIUM-99	5.33E-02 +- 2.1E-01	U	pCi/g	100.00%	3.09E-01	
9528-0003-019F	E94PN1AC	CARBON-14	-3.34E-02 +- 7.6E-02	U	pCi/g	100.00%	1.44E-01	
9528-0003-019F	E94PN1AM	AMERICIUM-241	2.00E-02 +- 2.1E-02	บ	pCi/g	79.23%	2.52E-02	
		CURIUM-242	0.00E+00 +- 2.3E-02	บ	pCi/g	79.23%	2.52E-02	
		CURIUM-243/244	0.00E+00 +- 1.3E-02	U	pCi/g	79.23%	1.45E-02	
9528-0003-019F	E94PN1AJ	NICKEL-63	2.28E+00 +- 5.6E+00	υ	pCi/g	77.20%	6.96E+00	

STL Richland

RER - Replicate Error Ratio = (S-D)/[sqrt(sq(TPUs)+sq(TPUd))] as defined by ICPT BOA.

rptSTLRchSaSum V3.97 A97 J Qual - No U qualifier has been assigned and the result is below the Reporting Limit, RL (CRDL) or Report Value is Estimated. U Qual - Analyzed for, but the result is less than the Mdc/Mda/Total Uncert or gamma scan software did not identify the nuclide.

# Sample Results Summary STL Richland STLRL

Ordered by Client Sample ID, Batch No.

Report No.: 21084

SDG No: 21324

Date: 30-Dec-02

Client ID	Work Order Number	Parameter	Result +- Uncertainty ( 2s)	Qual	Units	Yield	MDC MDA	RER
9528-0003-019F	E94PN1AK	IRON-55	3.78E+01 +- 1.7E+01	J	pCi/g	60.39%	2.66E+01	,
9528-0003-019F	E94PN2AF	STRONTIUM-90	4.70E-02 + 2.6E-02	J	pCi/g	48.12%	3.88E-02	
9528-0003-019F DUP	E94PN1AQ	NICKEL-63	1.79E+00 +- 4.9E+00	U	pCi/g	88.96%	6.01E+00	0.1
9528-0003-019F DUP	E94PN1AT	IRON-55	-1.08E+01 +- 1.9E+01	υ	pCl/g	42.63%	3.75E+01	3.8
9528-0004-008F DUP	E969G1AN	PLUTONIUM-238	-8.40E-04 +- 1.7E-03	U	pCi/g	78.83%	2.01E-02	0.2
	PLUTONIUM- 239/40	3.35E-02 +- 2.4E-02	J	pCi/g	78.83%	1.13E-02	0.7	
9528-0004-008F DUP	E969G1AP	PLUTONIUM-241	1.40E-01 +- 3.3E-01	U	pCi/g	78.80%	6.72E-01	0.7
9528-0004-010F DUP	E969N2AN	STRONTIUM-90	3.30E-02 +- 4.0E-02	U	pCi/g	37.02%	7.25E-02	0.2
Number of Results:	200							

<sup>-</sup> Replicate Error Ratio = (S-D)/[sqrt(sq(TPUs)+sq(TPUd))] as defined by ICPT BOA.

J Qual - No U qualifier has been assigned and the result is below the Reporting Limit, RL (CRDL) or Report Value is Estimated. U Qual - Analyzed for, but the result is less than the Mdc/Mda|Total Uncert or gamma scan software did not identify the nuclide.